



4-22221/A/DIV

March 2005

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of :  
Athanassios Tzikas et al :  
Serial No. 10/618,922 : Art Unit 1626  
Filed July 14, 2003 :  
For METHOD OF PRINTING CELLULOSIC FIBRE  
MATERIALS WITHOUT AN ADDITIONAL FIXING  
PROCESS STEP

DECLARATION

I, Georg ROENTGEN, chemical engineer of the Fachhochschule Aachen (Germany), a citizen of Germany, residing at Pochgasse 13, D-79104 Freiburg i. Br., hereby declare:

That I have been employed as a research chemist by Ciba Specialty Chemicals, Basel, since 1990;

That I have been engaged in the field of dyestuffs for Ciba Specialty Chemicals since 1990;

That based on the above education and experience, I consider myself an expert in the field of dyestuffs.

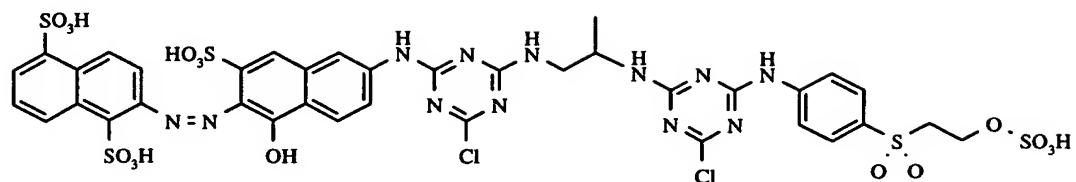
I, Georg ROENTGEN, declare that the following prints and tests were carried out under my direction and supervision;

That I am submitting herewith the following exact report of the tests mentioned below.

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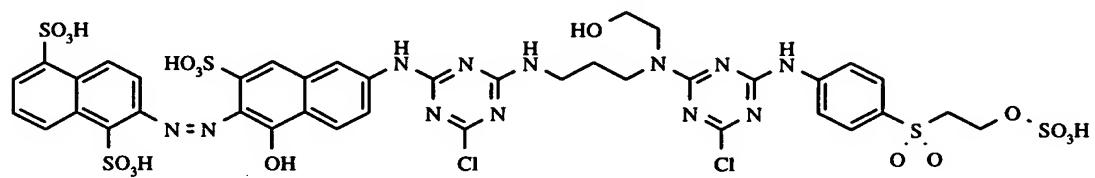
## 1. Preparation of dyestuffs

### Dyestuff A



according to the application, supra, obtained in analogy to the instructions given in Example 1 of US-A-5,989,297.

### Dyestuff B



according to US-A-5,989,297, obtained in analogy to the instructions given in Example 1 of US-A-5,989,297.

## 2. Printing of cotton satin

*Printing method according to column 46 of US-A-5,989,297*

1, 3 or 5 parts of dyestuff A (or dyestuff B) were sprinkled with high-speed stirring into 100 parts of stock thickening containing 50 parts of 5% strength sodium alginate thickening, 36.5 parts of water, 10 parts of urea, 1 part of sodium m-nitrobenzene sulfonate and 2.5 parts of sodium hydrogen carbonate. The print paste thus obtained was used to print a cotton satin fabric, which was then dried and the resulting printed fabric was steamed at 102°C in saturated steam for 8 minutes. The printed fabric was then rinsed and dried.

## 3. Build-up capacity

The tinctorial strength of the prints obtained according to 2. with the print pastes containing either dyestuff A or dyestuff B was measured photospectrometrically. The results are shown in the following Table 1, wherein the tinctorial strength of the prints is given in units of BZT (BZT is an internal measure for the tinctorial strength which is commensurate with the standard depth according to ISO/A-1984(E), page 4).

Table 1: Tinctorial strength (BZT) of the prints obtained according to 2.

parts of dyestuff	0	1.0	3.0	5.0
dyestuff A [BZT]	0	0.520	1.350	1.760
dyestuff B [BZT]	0	0.499	1.247	1.644

Table 1 demonstrates that the prior art dyestuff B builds up more poorly and yields a tinctorial strength in each case being inferior to dyestuff A according to the application, supra.

In other words, the concentration of prior art dyestuff B which is required to adjust a desired tinctorial strength is higher than the concentration of inventive dyestuff A. In order to adjust a tinctorial strength of 1.0 BZT 2.06 parts of inventive dyestuff A are required, whereas 2.23 parts of dyestuff B are required to achieve the same tinctorial strength.

Evidently, dyestuff A according to the application, supra, has a better affinity to cotton material in the printing process according to 2. than dyestuff B according to US-A-5,989,297.

#### 4. Washing-off properties

Cycle 1: A dried specimen of a printed cotton satin fabric with dyed and plain areas obtained according to the printing method 2. above (tinctorial strength: 1.0 BZT) was treated for 60 seconds at 98°C with an AHIBA-dyeing apparatus containing water (hardness: 20°dH) and 2.5 g/l of a softener (Calgon™). The corresponding bath after treatment is designated "bath 1". After the treatment the specimen was rinsed for 5 minutes with deionized water.

Cycle 2: Another dried specimen of the printed cotton satin fabric with dyed and plain areas obtained according to the printing method 2. was treated in bath 1 for 60 seconds at 98°C and afterwards in another freshly prepared bath for 60 seconds at 98°C. The corresponding bath after the treatment is designated "bath 2". After the treatment the specimen was rinsed for 5 minutes with deionized water.

Cycle 3: Still another dried specimen of a printed cotton satin fabric with dyed and plain areas obtained according to the printing method 2. was treated in bath 1 for 60 seconds at 98°C, afterwards in bath 2 for 60 seconds at 98°C and then in still another freshly prepared bath for 240 seconds at 98°C. After the treatment the specimen was rinsed for 5 minutes with deionized water.

Before each of the cycles 1 to 3 the printed cotton satin fabric was wound up and placed in a small metal basket for the washing-off treatment described above, so that printed and plain areas were in contact.

The washing-off test was developed in house in order to mimic the situation in a dyeing mill, wherein the printed textile fabric web is continuously guided through a water bath. During the washing-off process the amount of unfixed dyestuff increases in the water bath and staining of plain parts is going to increase.

Staining of plain areas of the printed specimen treated as above was assessed with the grey scale. In this regard, a high rating corresponds to a low amount of staining. Separate assessment was made for the inner part (inside) and the outer part (outside) of the wound specimen. The results are given in Table 2.

**Table 2:** Staining of plain areas of printed cotton satin fabric specimens

	Cycle 1 rating [grey scale]	Cycle 2 rating [grey scale]	Cycle 3 rating [grey scale]
Specimen printed with dyestuff A	4 (inside) 4 (outside)	4 (inside) 4 (outside)	4 (inside) 4 (outside)
Specimen printed with dyestuff B	4 (inside) 4 (outside)	3-4 (inside) 3-4 (outside)	3-4 (inside) 3-4 (outside)

Table 2 demonstrates that staining is less in the case of the specimens printed with inventive dyestuff A for cycles 2 and 3 (inside as well as outside).

### 5. Color fastness to water

The following test of color fastness to water was performed according to International Standard ISO 105-E01. Accordingly, an air dried specimen of the fabrics treated according to test methods 4. was sewn together with a piece of an undyed fibre material made of cotton and soaked in distilled water at room temperature. Excess water was poured off and the samples were then placed in a testing apparatus between two glass plates and burdened with a weight of 125 g/cm<sup>2</sup>. The testing apparatus containing the samples was warmed to 37°C and maintained at this temperature over a period of four hours. Finally, the samples were removed from the testing apparatus and dried in warm air.

Staining of undyed cotton fibre material received from the samples is assessed with the grey scale. In this regard, a high rating corresponds to a low amount of staining. Results are given in Table 3 below.

**Table 3:** Staining of an undyed cotton fibre material

	Cycle 1 rating [grey scale]	Cycle 2 rating [grey scale]	Cycle 3 rating [grey scale]
Specimen printed with dyestuff A	2-3 (inside) 3 (outside)	3 (inside) 3-4 (outside)	4 (inside) 4 (outside)
Specimen printed with dyestuff B	2-3 (inside) 2-3 (outside)	3 (inside) 3 (outside)	3-4 (inside) 4 (outside)

Table 3 demonstrates that staining is less in the case of the specimens printed with inventive dyestuff A for cycles 1 and 2 (outside) and cycle 3 (inside).

I, Georg ROENTGEN, hereby declare:

1. That based on my education and experience, I consider myself an expert in the field of dyeing art and dyestuff preparation;
2. That the results of the above tests show that the dyestuff according to the application, supra, is superior to the dyestuff according to the prior art with respect to the properties tested;
3. That build-up capacity, washing-off properties and fastness to water are important features for the textile industry and an improvement in these properties is of considerable importance;
4. That the above given tests demonstrate a significant improvement in these properties, which is of commercial importance;
5. That the improvement attainable with respect to build-up capacity, washing-off properties and fastness to water could not be foreseen and the results of the tests are surprising to me and I would never have predicted such difference in the property tested.

I, Georg ROENTGEN, declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 03<sup>rd</sup> day of March 2005



The signature consists of two parts: "Georg" on the left and "Roentgen" on the right, connected by a horizontal line. Below the signature, the name "Georg ROENTGEN" is printed in a standard font.